



AU9480459

(12) PATENT ABRIDGMENT (11) Document No. AU-B-80459/94  
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 674464

- (54) Title  
HERBICIDAL COMPOSITION CONTAINING GLYPHOSATE AND A C1-C6 ALKY AMMONIUM  
SULPHATE
- International Patent Classification(s)  
(51)<sup>6</sup> A01N 025/02 A01N 025/22 A01N 057/20
- (21) Application No. : 80459/94 (22) Application Date : 14.12.94
- (30) Priority Data
- (31) Number (32) Date (33) Country  
PM2933 14.12.93 AU AUSTRALIA
- (43) Publication Date : 22.06.95
- (44) Publication Date of Accepted Application : 19.12.96
- (71) Applicant(s)  
NUFARM LIMITED
- (72) Inventor(s)  
PHILLIP MAXWELL HAY
- (74) Attorney or Agent  
PHILLIPS ORMONDE & FITZPATRICK , 367 Collins Street, MELBOURNE VIC 3000
- (56) Prior Art Documents  
AU 595406 82045/87 A01N 57/20 59/02  
AU 54502/90 A01N 057/20
- (57) Claim

1. An aqueous herbicidal composition including glyphosate or a water soluble glyphosate salt and at least one alkyl-substituted ammonium sulphate selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate, di-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate and tri-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate.

6. A composition according to claim 1 wherein the total concentration of said glyphosate or water soluble glyphosate component is in the range of from 0.02 to 450 grams per litre (expresses as grams of glyphosate acid per litre), the total concentration of said alkyl substituted ammonium sulphate component is in the range of from 0.005 to 700 grams per litre and the molar ratio of said glyphosate or water soluble salt component to the alkyl substituted ammonium sulphate component is in the range of from 1:20 to 20:1.

(11) AU-B-80459/94  
(10) 674464

-2-

12. A process for preparation of a herbicidal composition according to claim 1 including forming a slurry of glyphosate in water acidifying the slurry with sulphuric acid and adding an alkylamine selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, di-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, tri-(C<sub>1</sub> to C<sub>6</sub>)alkylamine and mixtures of two or more alkylamines from one or more of these groups to provide an aqueous solution of alkylamine salt of glyphosate and alkylammonium sulphate.

**AUSTRALIA**

**Patents Act**

**COMPLETE SPECIFICATION  
(ORIGINAL)**

**Application Number:**  
**Lodged:**

**Class**

**Int. Class**

**Complete Specification Lodged:**  
**Accepted:**  
**Published:**

**Priority**

**Related Art:**

---

**Applicant(s):**

**NUFARM LIMITED**  
**103-105 Pipe Road**  
**Laverton North Victoria 3026**  
**Australia**

**Address for Service is:**

**PHILLIPS ORMONDE & FITZPATRICK**  
**Patent and Trade Mark Attorneys**  
**367 Collins Street**  
**Melbourne 3000 AUSTRALIA**

**Complete Specification for the invention entitled:**  
**" HERBICIDAL COMPOSITION "**

**Our Ref: IRN 338297**

**The following statement is a full description of this invention, including  
the best method of performing it known to applicant(s):**

# HERBICIDAL COMPOSITION

The invention relates to a herbicidal composition containing glyphosate or a salt thereof.

5

Glyphosate which is N-phosphonomethylglycine is generally used in the form of the isopropyl amine, ammonium or sodium salt.

10

Ammonium sulfate is known to improve the effectiveness of glyphosate by providing more rapid and more effective uptake of the herbicide, with consequently enhanced weed control and provides quicker knock-down of weeds. However, herbicidal formulations of glyphosate and ammonium sulfate in aqueous solution are unstable and tend to result in precipitation of the glyphosate salt. As a consequence mixtures of glyphosate and ammonium sulphate must generally be prepared shortly before use.

15

20

The present invention provides an aqueous herbicidal composition comprising glyphosate or a water soluble glyphosate salt and at least one alkyl-substituted ammonium sulfate selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)-alkyl ammonium sulfate, di-(C<sub>1</sub> to C<sub>6</sub>)-alkyl ammonium sulfate and tri-(C<sub>1</sub> to C<sub>6</sub>)-alkyl ammonium sulfate.

25

Most preferably the composition includes glyphosate or a water soluble glyphosate salt and a mono-(C<sub>1</sub> to C<sub>6</sub>)-alkyl ammonium sulfate, particularly mono-isopropylammonium sulfate.

30

Glyphosate is preferably present in the form of a water soluble alkyl-amine salt, particularly the mono(iso-propylamine) salt. It is particularly preferred that the alkylamine counter ion of the glyphosate is the same as the alkyl amine from which the alkylammonium sulphate is derived.

35

39

The concentration of glyphosate in the formulation is preferably in the range 0.02-450 grams per litre, expressed as grams glyphosate acid per litre. The concentration range of

the alkyl-substituted ammonium sulfate is preferably in the range 0.005-700 grams per litre. The molar ratio of glyphosate to the alkyl-substituted ammonium sulfate is typically in the range of from 1:20 to 20:1 and preferably from 1:10 to 10:1.

The composition of the invention may include one or more additional components such as surfactants, wetting agents, anti-foaming agents, anti-oxidants, thickening agents, humectants, dyes, adhesive/sticking agents and one or more other active agents which may be selected from herbicides, insecticides and/or fungicides. The additional components may be in the range of from 0 to 975 grams per litre.

The composition of the invention preferably contains one or more surfactants. Preferred surfactants are selected from the group consisting of alkylamine alkoxyates, trialkylamine oxides and quaternary ammonium compounds.

Examples of alkylamine alkoxyate surfactants include  $C_6$  to  $C_{30}$  alkylamines condensed with from 5 to 30 moles of an alkylene oxide (such as ethylene oxide or propylene oxide) per mole of amine. A specific example of an alkylamine alkoxyate surfactant is the compound formed by condensing a  $C_{16}$  alkylamine with 15 moles of ethylene oxide per mole of amine. Such a surfactant is sold under the trade mark TERIC 16M15 by ICI Australia.

Examples of trialkylamineoxides include fatty alkyl di( $C_1$  to  $C_6$ )alkylamine oxides such as lauryldimethylamine oxide.

Examples of quaternary ammonium surfactants include the quaternary ammonium compounds including at least one  $C_6$  to  $C_{30}$  alkyl group.

The composition of the invention may be prepared at a wide range of concentrations as hereinabove described. This enables the composition to be prepared as stable concentrate for storage and transport and to be diluted prior to use.

In a preferred embodiment we therefore provide a herbicidal concentrate comprising glyphosate or a water soluble glyphosate salt in an amount of from 20 to 450g per litre, expressed as grams glyphosate acid per litre, alkyl substituted ammonium sulphate in the range of 5 to 700 grams per litre, optionally additional components in an amount of from 0 to 975 grams per litre, more preferably 0.1 to 400 grams per litre and water from 100 to 950 g/litre.

On a weight basis the composition preferably comprises glyphosate or a water soluble salt of glyphosate in the range of 2 to 60% by weight preferably 20 to 60% (based on the weight of free acid), alkylammonium sulphate in the range of 0.5 to 70% by weight, an alkylamine in the range of 0.5 to 70%, a surfactant in the range of from 0.001 to 15% and water in an amount of 10 to 90% by weight. Other optional additives may be present, for example, in the range of from 0.001 to 60%.

The herbicidal composition of the invention may be prepared by simply combining the components, that is the glyphosate or water soluble glyphosate salt and alkylamine sulphate, water and any other components however it is particularly preferred to prepare the alkylammonium sulphate in situ by reaction of an alkylamine and sulphuric acid. In a further aspect of the invention provides a method for the preparation of the hereinabove described herbicidal composition including reacting sulphuric acid and alkylamine in the presence of glyphosate in the form of the glyphosate acid or water soluble glyphosate salt to form a mixture including glyphosate acid or water soluble salt and alkyl ammonium sulphate. Preferably the glyphosate is reacted with the alkylamine to form an alkylamine glyphosate salt.

The alkylamine is a mono-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, a di(C<sub>1</sub> to C<sub>6</sub>)alkylamine, a tri(C<sub>1</sub> to C<sub>6</sub>)alkylamine or a mixture of two or more alkylamines selected from one or more of these classes of alkylamines.

The glyphosate may, as a preliminary step, be dissolved in an alkylamine or it may simply be slurried in water.

5 In a preferred embodiment an aqueous slurry of glyphosate is formed the slurry is acidified (preferably to provide a pH of less than 2) using sulphuric acid (preferably concentrated sulphuric acid) and the alkylamine is then added to provide a pH preferably in the range of 3.5 to 6 and to form an aqueous mixture of alkylamine salt of glyphosate and alkylammonium sulphate.  
10

Alternatively glyphosate acid or glyphosate water soluble salt is dissolved to form an alkylamine salt of glyphosate and the mixture acidified by addition of aqueous sulphuric acid to provide the alkylammonium sulphate.  
15

In a further alternative method the composition is prepared by providing an aqueous solution of sulphuric acid, reacting the sulphuric acid with an alkylamine to provide an aqueous mixture of alkylamine sulphate and mixing glyphosate with the composition, optionally with further addition of alkylamine, to form an aqueous solution of alkylammonium sulphate and the alkylamine salt of glyphosate.  
20

The composition of the invention may be prepared at a range of temperatures for example in the range of 5 to 95°C however ambient temperature is typically convenient.  
25

The composition of the invention will preferably be applied to crops at rate of from 240 to 3240 gram per hectare, expressed on the basis of glyphosate acid. In accordance with a further aspect the invention therefore provides a method for controlling weeds comprising applying to the locus of the weeds a formulation as herein described at rate in the range of from 240 to 3240 gram per hectare, expressed on the basis of glyphosate acid.  
30  
35

The glyphosate may, as a preliminary step, be dissolved in an alkylamine or it may simply be slurried in water.

5 In a preferred embodiment an aqueous slurry of glyphosate is formed the slurry is acidified (preferably to provide a pH of less than 2) using sulphuric acid (preferably concentrated sulphuric acid) and the alkylamine is then added to provide a pH preferably in the range of 3.5 to 6 and to form an aqueous mixture of alkylamine salt of glyphosate and alkylammonium sulphate.  
10

Alternatively glyphosate acid or glyphosate water soluble salt is dissolved to form an alkylamine salt of glyphosate and the mixture acidified by addition of aqueous sulphuric acid to provide the alkylammonium sulphate.  
15

In a further alternative method the composition is prepared by providing an aqueous solution of sulphuric acid, reacting the sulphuric acid with an alkylamine to provide an aqueous mixture of alkylamine sulphate and mixing glyphosate with the composition, optionally with further addition of alkylamine, to form an aqueous solution of alkylammonium sulphate and the alkylamine salt of glyphosate.  
20

The composition of the invention may be prepared at a range of temperatures for example in the range of 5 to 95°C however ambient temperature is typically convenient.  
25

The composition of the invention will preferably be applied to crops at rate of from 240 to 3240 gram per hectare, expressed on the basis of glyphosate acid. In accordance with a further aspect the invention therefore provides a method for controlling weeds comprising applying to the locus of the weeds a formulation as herein described at rate in the range of from 240 to 3240 gram per hectare, expressed on the basis of glyphosate acid.  
30  
35



#### EXAMPLE 1

A composition of the invention containing glyphosate isopropylamine salt (120 g/l based on glyphosate acid) was prepared by the following process involving formation of the glyphosate salt and isopropylammonium sulphate in situ.

126g of 95% purity glyphosate acid was slurried in 300 mL water and 50g TERIC 15M5 surfactant. 210g concentrated sulfuric acid was slowly added to the slurry with good agitation. The acidified slurry was then neutralised to pH 4.5 - 5.5 with isopropylamine (480g of 60% aqueous solution). The mixture was then diluted to 1 L with water.

The composition was found to have good storage stability with no appreciable precipitation of glyphosate salts.

#### EXAMPLE 2

The following components were used in preparing a herbicidal composition using the same general procedure as described in Example 1.

	g	% w/w total
technical glyphosate (95% w/w purity on dry basis, 3% w/w water)	130.3	9.3
sulfuric acid (98% w/w)	210	15
iso-propylamine (60% w/w)	482.9	34.5
C <sub>16-18</sub> primary fatty amine ethoxylate (15 mol EO/mol)	52	3.7
water	525	37.5
Total	1400	

EXAMPLE 3

A trial to evaluate the efficacy of a formulation of the invention against *Ottochloa nodosa* under 0% shade in an oil palm plantation in Malaysia was conducted as follows:

(a) A composition of the invention ("Trial") was prepared in accordance with the general procedure of Example 1 having the composition described in Table 1 ("Trial"). Two other compositions one (referred to as "comparison") containing glyphosate, ammonium sulfate and a surfactant and the other (a proprietary formulation) containing glyphosate ammonium sulphate and an undisclosed surfactant as shown in Table 1. In each case the glyphosate is present as the isopropylamine salt.

Table 1

Component	Trial g/L	Comparison g/L	Commercial Preparation g/L
*glyphosate	120	120	120
TERIC 16M15 surfactant	50	50	-
proprietary surfactant			not disclosed
ammonium sulphate	-	270	317
isopropyl amine sulfate	425	-	-
water	to volume	to volume	

\* Weights of glyphosate isopropylamine are given on the basis of the weight of free acid.

The compositions were tested on plots of oil palm crop having

a uniform 85% weed cover at the commencement of the trial. The weed species present was predominantly Ottachloa nodosa. Two dilution rates were tested as described in (b) and (c) below.

5

(b) Compositions were prepared for spraying by diluting 2 litres of concentrate to 225 litres with water and applying the diluted composition at a rate of 225 litres per hectare.

10

Table 2

240g glyphosate per hectare

15

	% WEED CONTROL			
	Trial	Comparison	Commercial	NOT TREATED
0 d.a.t	0	0	0	0
14 d.a.t	73	73	63	0
21 d.a.t	87	82	77	0
30 d.a.t	83	72	70	0

20

where d.a.t. represents days after treatment

(c) Compositions were prepared for spraying by diluting 4 litres of concentrate to 225 litres with water and applying the diluted composition at the rate of 225 litres per hectare. The results are shown in Table 3.

25

Table 3

480g glyphosate per hectare

35

	% WEED CONTROL			
	Trial	Comparison	Commercial	NOT TREATED
0 d.a.t	0	0	0	0
14 d.a.t	78	80	78	0
21 d.a.t	90	92	92	0
30 d.a.t	90	89	93	0

39

where d.a.t. represents days after treatment.

5 The test shows that at low application rates the composition  
of the invention shows significantly improved weed control.

10 Finally, it is to be understood that various other  
modifications and/or alterations may be made without departing  
from the spirit of the present invention as outlined herein.

15

20

25

30

35

39

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An aqueous herbicidal composition including glyphosate or a water soluble glyphosate salt and at least one  
5 alkyl-substituted ammonium sulphate selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate, di-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate and tri-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate.
- 10 2. A composition according to claim 1 wherein the alkyl substituted ammonium sulphate is a mono-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate.
- 15 3. A composition according to claim 1 wherein the alkyl-substituted ammonium sulphate is isopropylammonium sulphate.
4. A composition according to claim 1 wherein said  
glyphosate or water soluble salt component includes a water  
soluble (C<sub>1</sub> to C<sub>6</sub>)-alkylamine salt of glyphosate.  
20 5. A composition according to claim 4 wherein the salt  
of glyphosate is the mono(isopropylamine) salt.
- 25 6. A composition according to claim 1 wherein the total  
concentration of said glyphosate or water soluble glyphosate  
component is in the range of from 0.02 to 450 grams per litre  
(expresses as grams of glyphosate acid per litre), the total  
concentration of said alkyl substituted ammonium sulphate  
component is in the range of from 0.005 to 700 grams per litre  
and the molar ratio of said glyphosate or water soluble salt  
30 component to the alkyl substituted ammonium sulphate component  
is in the range of from 1:20 to 20:1.
- 35 7. A composition according to claim 1 wherein the  
composition includes one or more surfactants selected from the  
group consisting of alkylamine alkoxyates, trialkylamine  
oxides and quaternary ammonium compounds.
- 39 8. A composition according to claim 7 wherein the

composition includes a surfactant selected from (C<sub>6</sub> to C<sub>30</sub> alkylamines condensed with from 5 to 30 moles of an alkylene oxide, di(C<sub>1</sub> to C<sub>6</sub>)alkylamine oxides and quaternary ammonium surfactants including at least one C<sub>6</sub> to C<sub>30</sub> alkyl group.

9. A composition according to claim 1 comprising glyphosate or a water soluble glyphosate salt in an amount of from 20 to 450 grams per litre, (expressed as grams of glyphosate acid per litre), a mono C<sub>1</sub> to C<sub>6</sub> alkylammonium sulphate in the range of 5 to 700 grams per litre, water in an amount of from 100 to 950 grams per litre and optionally additional components in an amount of from 0 to 975 grams per litre.

10. A composition according to claim 1 wherein the alkylammonium sulphate component is prepared in situ by reacting an alkylamine selected from a mono-(C<sub>1</sub> to C<sub>6</sub>) alkylamine, a di-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, a tri-(C<sub>1</sub> to C<sub>6</sub>)alkylamine or mixtures of two or more alkylamines selected from one or more of these classes of alkylamines with sulphuric acid.

11. A composition according to claim 10 wherein the alkylamine is a mono-(C<sub>1</sub> to C<sub>6</sub>)-alkylamine.

12. A process for preparation of a herbicidal composition according to claim 1 including forming a slurry of glyphosate in water acidifying the slurry with sulphuric acid and adding an alkylamine selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, di-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, tri-(C<sub>1</sub> to C<sub>6</sub>)alkylamine and mixtures of two or more alkylamines from one or more of these groups to provide an aqueous solution of alkylamine salt of glyphosate and alkylammonium sulphate.

13. A process for preparation of a herbicidal composition according to claim 1 comprising dissolving glyphosate in an alkylamine selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, di-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, tri-(C<sub>1</sub> to

C<sub>6</sub>)alkylamine and mixtures of two or more alkylamines from one or more of these groups to form the alkylamine salt of glyphosate and adding aqueous sulphuric acid optionally with further addition of alkylamine, to form an aqueous solution of alkylammonium sulphate and alkylamine salt of glyphosate.

14. A process for preparation of a herbicidal composition according to claim 1 comprising providing an aqueous sulphuric acid solution, reacting the sulphuric acid with an alkylamine selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>) alkylamine, di-(C<sub>1</sub> to C<sub>6</sub>)alkylamine, tri-(C<sub>1</sub> to C<sub>6</sub>) alkylamine and mixtures of two or more alkylamines from one or more of these groups to provide an aqueous mixture of alkylamine sulphate and mixing glyphosate with the composition, optionally with further addition of alkylamine, to form an aqueous solution of alkylamine sulphate and the alkylamine salt of glyphosate.

15. A process for treating weeds in a crop including applying to the locus of the weeds a herbicidal composition according to any one of claims 1 to 12.

16. A process according to claim 15 wherein the crop is palm oil.

17. A composition according to claim 16 substantially as herein described with reference to any one of the examples.

DATED: 14 December 1994

PHILLIPS ORMONDE & FITZPATRICK  
Attorneys for:  
NUFARM LIMITED

*David B Fitzpatrick*

3297S

# ABSTRACT

An aqueous herbicidal composition including glyphosate or a water soluble glyphosate salt and at least one alkyl-substituted ammonium sulphate selected from the group consisting of mono-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate, di-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate and tri-(C<sub>1</sub> to C<sub>6</sub>)-alkylammonium sulphate.

10

15

20

25

30

35

39